## Appendix F

## SEM/EDS Data for Test #3, Day-30 Flow Meter

### **Figures**

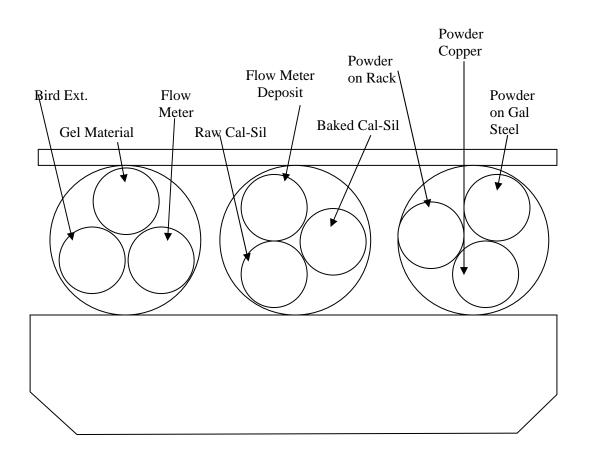
Figure F-1.	SEM image magnified 80 times for Test #3, Day-30 debris within the	
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	shown in Figure F-4. (T3Deposits08)	F-7
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In ICET Test #3, significant amounts of debris and precipitates were found within the flow meter. SEM/EDS analysis was performed to examine the composition of the debris trapped in the flow meter, as well as the white precipitates deposited on the inner wall of the flow meter. The debris and the precipitates were collected on May 5, 2005, the date Test #3 was shut down. The samples were dried in air before being coated with Au/Pd for SEM examination. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

#### **Transcribed Laboratory Log**

#### Laboratory session from May 9, 2005.

Test #3, Day-30 Flow Meter



#### **Flow Meter Debris**

Image:	T3D30FlwMetrDebris005	80 ×	Figure F-1
	T3D30FlwMetrDebris006	600 ×	Figure F-2
EDS:	T3D30FlwDebris03		Figure F-3

### **Flow Meter Deposits**

Image:	T3~Flow Meter	200 ×	Figure F-4
EDS:	T3Deposits08		Figure F-5

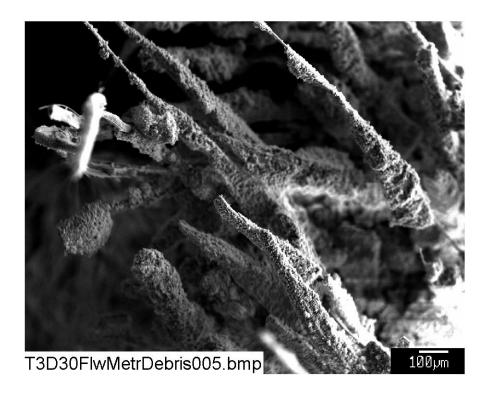


Figure F-1. SEM image magnified 80 times for Test #3, Day-30 debris within the flow meter. (T3D30FlwMetrDebris005)

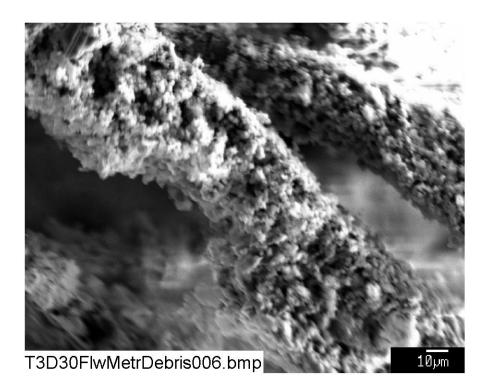


Figure F-2. SEM image magnified 600 times for Test #3, Day-30 debris within the flow meter. (T3D30FlwMetrDebris006)

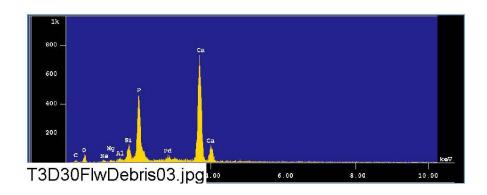


Figure F-3. EDS counting spectrum for the coatings on the fibers shown in Figure F-2. (T3D30FlwDebris03)

The results from the chemical composition analysis for T3D30FlwDebris03 are given in Table F-1.

Chemical Compositions for T3D30FlwDebris03, Figure F-3

#### May 9 2005

: NRC Group Sample : T3D30 ID# : 3 Comment : Flowmeter Debris Condition : Full Scale : 20KeV(10eV/ch,2Kch)

Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.596E-09 A
Stage Point : X=77.422 Y=68.992 Z=12.516

Acq. Date : Mon May 9 12:10:19 2005

Elemen	nt Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Backgrou	und
ок	Normal	0.25- 0.77	1.1291	0.0010	422 /	12
Si K	Normal	1.50- 2.05	0.6220	0.0004	899 /	142
PΚ	Normal	1.75- 2.38	4.6152	0.0040	4117 /	58
Ca K	Normal	3.39- 4.30	12.9947	0.0033	9129 /	16
CK	Normal	0.09- 0.46	0.1025	0.0001	57 /	9
Pd L	Normal	2.22- 3.81	0.5748	0.0010	419 /	35
Al K	Normal	1.19- 1.83	0.0834	0.0002	129 /	24
Na K	Normal	0.81- 1.27	0.0806	0.0004	86 /	10
Mg K	Normal	0.97- 1.57	0.0536	0.0001	84 /	15

Chi\_square = 8.9413

Element	Mass*	Atomic%	ZAF	$\mathbf{z}$	A	F	
0	15.028	28.4942	2.9323	0.9460	3.0996	1.0000	
Si	3.174	3.4286	1.1244	0.9475	1.1960	0.9922	
P	17.384	17.0260	0.8298	1.1366	0.7330	0.9960	
Ca	57.802	43.7487	0.9799	0.9630	1.0176	1.0000	
C	1.833	4.6300	3.9385	0.9925	3.9689	0.9999	
Pd	3.395	0.9679	1.3011	1.3345	0.9949	0.9799	
Al	0.487	0.5473	1.2851	0.9696	1.3304	0.9962	
Na	0.538	0.7099	1.4707	0.9973	1.4750	0.9999	
Mg	0.359	0.4474	1.4751	0.9384	1.5740	0.9987	

Total 100.000 100.0000

Normalization factor = 4.5392

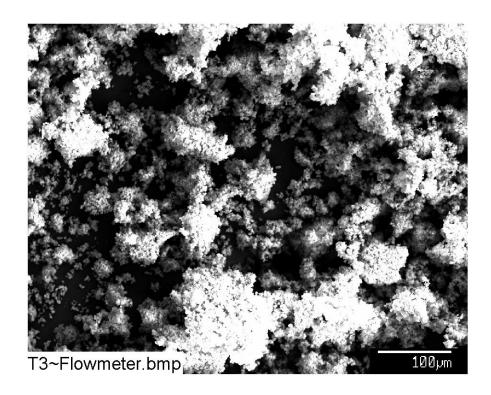


Figure F-4. SEM image magnified 200 times for Test #3, Day-30 deposits on the inner wall of the flow meter. (T3~Flowmeter)

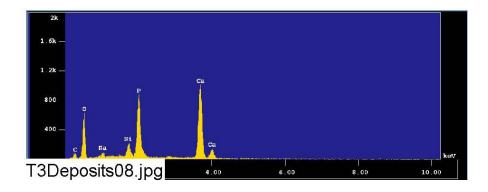


Figure F-5. EDS counting spectrum for the large masses of particulate deposits shown in Figure F-4. (T3Deposits08)

The results from the chemical composition analysis for T3Deposits08 are given in Table F-2.

Table F-2. Chemical Compositions for T3Deposits08, Figure F-5

#### May 9 2005

: NRC Group Sample : T3D30 ID# : 8 Comment : Flowmeter Deposits

Condition: Full Scale: 20KeV(10eV/ch,2Kch)

Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.607E-09 A
Stage Point : X=47.897 Y=71.447 Z=12.516

Acq. Date : Mon May 9 14:45:11 2005

Eler	ment	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Back	ground
C	K	Normal	0.09- 0.46	0.5235	0.0004	292	/ 100
0	K	Normal	0.25- 0.77	10.8619	0.0030	4085	/ 54
Na	K	Normal	0.81- 1.27	0.2843	0.0009	307	/ 44
Si	K	Normal	1.50- 2.05	1.0559	0.0005	1537	/ 258
P	K	Normal	1.75- 2.38	8.7448	0.0054	7854	/ 123
Ca	K	Normal	3.39- 4.30	17.8554	0.0039	12630	/ 21

Chi square = 35.5886

43.460	7.2812 60.3090					
	60.3090	2.0251	0 9706	0 0065		
			0.3700	2.0865	1.0000	
0.842	0.8134	1.4997	1.0240	1.4645	1.0000	
2.376	1.8782	1.1389	0.9739	1.1772	0.9933	
14.513	10.4028	0.8400	1.1688	0.7203	0.9976	
34.870	19.3155	0.9884	0.9928	0.9956	1.0000	
]	2.376 14.513	2.376 1.8782 14.513 10.4028	2.376 1.8782 1.1389 14.513 10.4028 0.8400	2.376 1.8782 1.1389 0.9739 14.513 10.4028 0.8400 1.1688	2.376	111111111111111111111111111111111111111

Total 100.000 100.0000

Normalization factor = 1.9758

Total 100.000 100.0000

Normalization factor = 2.1120

### Appendix G

## SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Gel

### **Figures**

Figure G-1.	SEM image magnified 100 times for a Test #3, Day-30 white gel-like
	material on the top of the birdcage. (T3D30GelMaterial003)
Figure G-2.	SEM image magnified 1000 times for a Test #3, Day-30 white gel-like
	material on the top of the birdcage. (T3D30GelMaterial004) G-5
Figure G-3.	EDS counting spectrum for the white gel-like material (whole image)
	shown in Figure G-2. (T3D30Gel02)
Figure G-4.	ESEM image magnified 1000 times for a Test #3, Day-30 white gel-like
	material on the top of the birdcage. (t3Gel08)
Figure G-5.	EDS counting spectrum for the white gel-like material shown in Figure
	G-4. (t3GelED4)
Figure G-6.	Comparison of EDS counting spectra for Figure G-5 (red, the gel-like
	materials shown in Figure G-4) and Figure C4-5 (yellow, the large deposits
	taken from the birdcage exterior shown in Figure C4-4). (t3geled5) G-9
Figure G-7.	Another EDS counting spectrum for the white gel-like material shown in
	Figure G-4. (t3geled6)
Figure G-8.	Comparison of EDS counting spectra for Figure G-7 (yellow, the gel-like
	materials shown in Figure G-4) and Figure C4-5 (red, the large deposits
	taken from the birdcage exterior shown in Figure C4-4). (t3geled7) G-10
Figure G-9.	XRD results for Test #3, Day-30 white gel-like material

### **Tables**

Table G-1.	Chemical Compositions for T3D30Gel02, Figure G-3	G-7
Table G-2.	Dry Mass Composition of a Test #3, Day 30 White Gel-Like Sample by XRF	
	Analysis	G-11

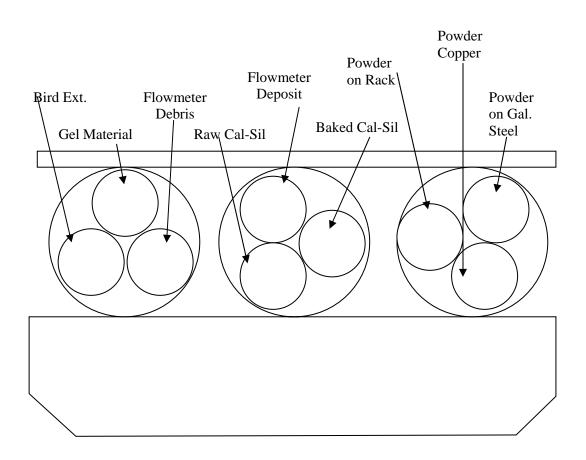
In ICET Test #3, one distinguished phenomenon is the presence of white gel-like precipitates in the testing solution. On the shutdown date of Test #3, deposits of the white gel-like precipitates were observed on the top of the birdcage. These precipitates may increase the containment sump screen head loss during a LOCA; therefore, it is necessary to investigate the morphology and composition of the white gel-like precipitate.

This appendix shows the ESEM/SEM/EDS and XRD/XRF results of the white gel-like precipitates. The precipitates were collected on the date Test #3 was shut down (May 5, 2005). For the SEM examination, the samples were dried in air before being coated with Au/Pd. EDS results provide a semi-quantitative elemental analysis of the sample compositions. Also, XRD results show the crystal structure of the white gel-like precipitates. Based on the XRD results, the composition of the white gel-like precipitates contained crystalline substances of sodium calcium hydrogen carbonate phosphate hydrate [Ca<sub>8</sub>H<sub>2</sub>(PO<sub>4</sub>)<sub>6</sub>·H<sub>2</sub>O·NaHCO<sub>3</sub>·H<sub>2</sub>O] and lithium calcium hydrogen carbonate phosphate hydrate [Ca<sub>8</sub>H<sub>2</sub>(PO<sub>4</sub>)<sub>6</sub>·H<sub>2</sub>O·Li<sub>2</sub>CO<sub>3</sub>·H<sub>2</sub>O]. In addition, XRF results indicate the chemical composition of the precipitates. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

### **Transcribed Laboratory Log**

### Laboratory session from May 9, 2005.

Test #3, Day-30 Gel Material

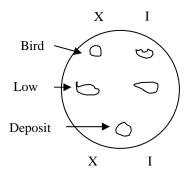


#### **Gel Material**

Image:	T3D30GelMaterial003	$100 \times$		Figure G-1
	T3D30GelMaterial004	1000 ×		Figure G-2
EDS:	T3D30Gel02		Whole screen of image 004	Figure G-3

### **Transcribed Laboratory Log**

# <u>Laboratory session from May 6, 2005.</u> Test #3, Day-30 Gel Material



### Gel-Like Material on Top of Birdcage

Image:	t3Gel08	1000 ×		Figure G-4
EDS:	t3GelED4		White gel has high C and low Si	Figure G-5
	t3geled5		Comparing t3bcexe2 from Appendix C4 & t3GelED4	Figure G-6
	t3geled6		EDS of gel material	Figure G-7
	t3geled7		Comparing t3bcexe2 from Appendix C4 & EDS6	Figure G-8

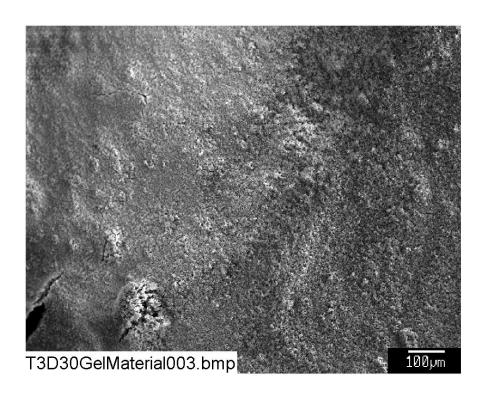


Figure G-1. SEM image magnified 100 times for a Test #3, Day-30 white gel-like material on the top of the birdcage. (T3D30GelMaterial003)

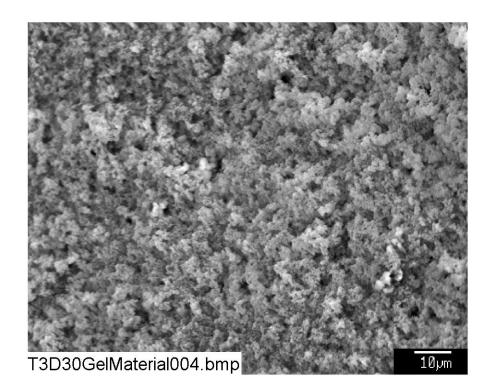


Figure G-2. SEM image magnified 1000 times for a Test #3, Day-30 white gel-like material on the top of the birdcage. (T3D30GelMaterial004)

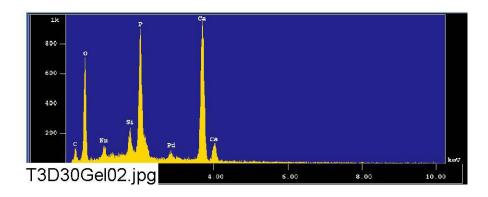


Figure G-3. EDS counting spectrum for the white gel-like material (whole image) shown in Figure G-2. (T3D30Gel02)

The results from the chemical composition analysis for T3D30Gel02 are given in Table G-1.

Table G-1. Chemical Compositions for T3D30Gel02, Figure G-3

#### May 9 2005

```
Group
          : NRC
Sample
         : T3D30 ID# : 2
Comment
         : GelMaterial
Condition: Full Scale: 20KeV(10eV/ch,2Kch)
            Live Time
Acc. Volt
                       : 60.000 sec Aperture #
                       : 15.0 KV
                                        Probe Current: 1.606E-09 A
            Stage Point : X=79.625 Y=59.260 Z=11.424
            Acq. Date
                        : Mon May 9 11:42:11 2005
Element
            Mode
                    ROI (KeV)
                                K-ratio(%) +/-
                                                   Net/Background
  CK
           Normal
                                                      338 /
                    0.09- 0.46
                                 0.6057
                                           0.0005
                                                                 119
  OK
                    0.25- 0.77
           Normal
                                 12.2043
                                           0.0032
                                                      4587 /
                                                                 68
 Na K
           Normal
                    0.81- 1.27
                                 0.5675
                                                      613 /
                                           0.0010
                                                                 50
 Si K
           Normal
                    1.50- 2.05
                                                      1366 /
                                 0.9391
                                           0.0005
                                                                 271
  PK
                    1.75- 2.38
           Normal
                                 8.4975
                                           0.0055
                                                      7628 /
                                                                 107
 Ca K
           Normal
                    3.39- 4.30
                                17.1295
                                           0.0038
                                                     12109 /
                                                                  26
                             Chi_square = 42.7915
Element Mass%
                Atomic*
                           ZAF
     C
          4.355
                7.8616 3.7318 1.0194 3.6611 0.9999
     0
         45.521 61.6928 1.9361 0.9721 1.9917 1.0000
    Na
          1.639
                1.5456 1.4989 1.0256 1.4614 1.0000
    Si
         2.072
                1.5994 1.1451 0.9756 1.1812 0.9937
    P
         13.776
                 9.6435 0.8415 1.1708 0.7203 0.9978
         32.638 17.6571 0.9890 0.9947 0.9943 1.0000
    Ca
Total 100.000 100.0000
Normalization factor = 1.9265
TOTAL 100.000 100.0000
Normalization factor = 2.1120
```

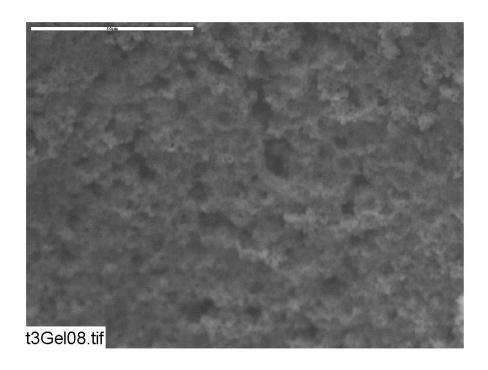


Figure G-4. ESEM image magnified 1000 times for a Test #3, Day-30 white gel-like material on the top of the birdcage. (t3Gel08)

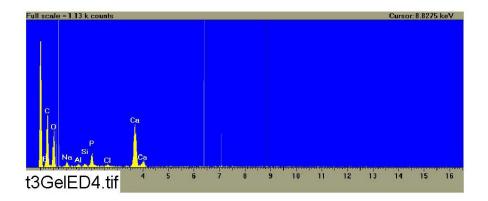


Figure G-5. EDS counting spectrum for the white gel-like material shown in Figure G-4. (t3GelED4)

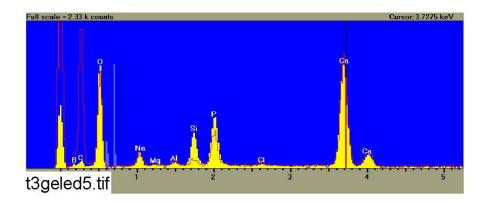


Figure G-6. Comparison of EDS counting spectra for Figure G-5 (red, the gel-like materials shown in Figure G-4) and Figure C4-5 (yellow, the large deposits taken from the birdcage exterior shown in Figure C4-4). (t3geled5)

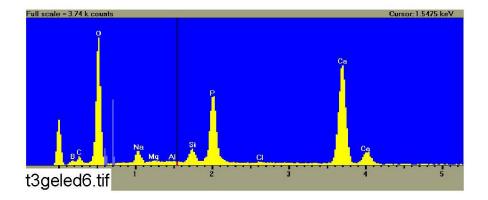


Figure G-7. Another EDS counting spectrum for the white gel-like material shown in Figure G-4. (t3geled6)

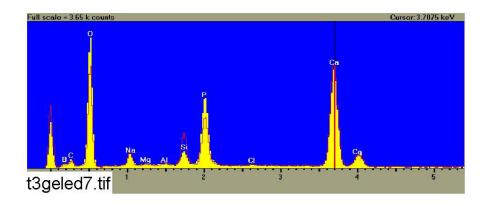


Figure G-8. Comparison of EDS counting spectra for Figure G-7 (yellow, the gel-like materials shown in Figure G-4) and Figure C4-5 (red, the large deposits taken from the birdcage exterior shown in Figure C4-4). (t3geled7)

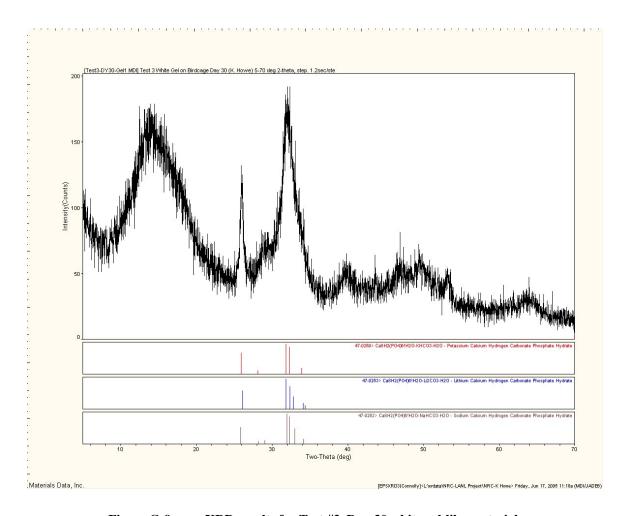


Figure G-9. XRD results for Test #3, Day-30 white gel-like material.

Table G-2. Dry Mass Composition of a Test #3, Day 30 White Gel-Like Sample by XRF Analysis

Sample ID	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	FeO	MnO	MgO		Na <sub>2</sub>	K <sub>2</sub> O		H <sub>2</sub> O(+) CO <sub>2</sub>	$P_2O_5$		H2O(+)CO2 /DF (10) & Cover. To %
<b>Test #3</b> ,															
<b>Day 30</b>															
White															
Gel	5.26	0.02	0.63	0.07	0.00	0.00	0.25	35.01	2.39	0.06	4.75	19.24	27.09	94.77	1.0196